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Development of a Large Sample Collector of Respirable Particulate Matter

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A prototype sampler designed to collect particulate matter from air in sized fractions has been designed and tested. The sampler excludes particles above 20 μm in diameter and collects fractions centered at 3.5 μm and 1.7 μm on impaction plates and smaller particles by electrostatic precipitation. The report includes test data, engineering drawings and materials list.

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DEVELOPMENT OF A LARGE SAMPLE COLLECTOR OF RESPIRABLE PARTICULATE MATTER

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ABSTRACT

This report presents the final results of an experimental program to develop a system of size-selective air particulate collection and analysis to provide compositional particulate air pollutant data in support of health and toxicity studies.

A massive air volume sampler has been designed and constructed to collect large masses of particulates in three particle size ranges: >3.5 μm, 1.7 to 3.5 μm, and <1.7 μm. The latter two smaller size ranges represent the particulates deemed by aerosol physiologists to be representative of the respirable size particles. The partition of the respirable fraction into two size ranges is to provide more definitive data on aerosols which have been created by secondary processes, such as photochemical reactions.

Field tests of the sampler design and construction show that better than gram masses of urban atmospheric particulate loadings can be collected in 24-hour periods; the samples can be recovered readily from the sampler collector substrates; and the mass fraction obtained correlates closely with cascade impactor sampling results.

Compositional and structural examinations of the collected particulates clearly show that different size ranges have different chemical and physical properties. It is foreseen that the mass collection capability of the sampler would provide respirable size particulate samples for certain types of health/toxicity studies as well as providing samples for chemical analysis.

Evaluations of the samplers in regard to sampling precision, collection efficiency, and possible reaction of sulfur dioxide and nitrogen dioxide during the collection process need completion. These efforts were interrupted

by a need to place the sampler immediately in the field at an Environmental Protection Agency background catalyst study site.

The field use of the samplers on a continuous basis, rather than in the intermittent mode as originally planned, revealed certain operational and stability problems. These problems were resolved under an expansion of the program efforts to provide for better structural and field operational performance.

This report is submitted in fulfillment of Contract No. 68-02-0752 by Battelle's Columbus Laboratories under the sponsorship of the U.S. Environmental Protection Agency. It covers a period from June 14, 1973 to April 13, 1975.

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SECTION I

CONCLUSIONS

Considerations of the requisite sample loading and size separation requirements of the program showed that a sampler design utilizing two impactor stages and an electrostatic precipitator could provide for collection of gram quantities of respirable range, urban air particulates in a 24-hour period.

Calibration and field test data demonstrated that the designed and constructed sampler does provide the size range cutoff particulate sizes deemed by aerosol physiologists as most representative for health effects study.

The collections can be readily removed from the Teflon-coated impactor stages and from the very pure aluminum (Al) electrostatic plates without introducing substrate contamination into the samples.

The sampler provides for two cutoff sizes in the respirable size range, 1.7 to 3.5 μm and <1.7 μm, and for particles above the respirable range, >3.5 to 20 μm. Thus, a total sample analysis can be obtained for comparison with the more prevalent total suspended particulate (TSP) data.

Collections of well over 1 gram (g) can be made in a 24-hour period, so detailed analytical methodology development can be carried out to provide compound and species identification needed for toxicological testing and for interrelating population health effects studies with particulate burdens. Analytical examinations show definite composition versus size relationships for many species.

In summary, the program efforts show the following benefits to health study programs.

(1) Realistic, "live", contaminant-free, air particulate burden samples can be collected for the effective carrying out of dose/response health effects surveys and studies. Realistic and "live" samples refer to particulates collected in large masses and segregated into respirable and nonrespirable cuts. These cuts show distinct differences in appearance and odor emanations. These physical attributes of particulate collections are striking and usually are masked when samples are imbedded in filter or other substrate modes of collection.

(2) The samplers can collect sized particulates in sufficient masses for in-vitro and in-vivo health studies.

(3) Samples can be collected in time periods which permit unusual atmospheric burden episodes, such as inversions or unusual source emissions, to be sampled, analyzed, and the particulate species related to observed health consequences. (Compare collection rates of about 1000 cfm with the 40 cfm of high volume [hi-vol] samplers.)

(4) Long-needed data bases can be obtained now on the presence and quantities of pollutant species contained in fine, respirable particulates. To date, overall species characterization efforts have been seriously hampered by lack of adequate, contaminant-free samples, with a result that comparatively little is known on the presence of overall specific and possible harmful compound forms of pollutants.

(5) Continued investigations, as outlined in the Recommendations, should permit completion of the original objective of the program: to identify and quantitate species implicated as causative agents for observed adverse health effects in man.

SECTION II

RECOMMENDATIONS

The design and construction efforts on this program have provided a novel system wherein airborne particulates can be collected in masses large enough both to obtain detailed knowledge of size-related chemical and physical properties of pollutants and their associated health effects via in-vitro and in-vivo studies. The applications recommended for use of the system include:

- Obtain short-term "episodic" respirable particulate compositional analysis for correlation with health effects studies at Community Health and Environmental Surveillance System (CHESS) sites.
- Monitor point sources upstream and downstream to ascertain potentially hazardous compound species emissions.
- Collect specific particles--asbestos, silica, radionuclides, etc.--on a size basis either in short- or long-term periods.
- Collect sized samples solely for health burden studies
- Couple the Massive Air Volume Sampler with gaseous pollutant sampling collection to obtain total respirable range health burdens of pollutants by urban area, source emission, industrial operation (mining, refining, processing), residential (indoor-outdoor), and/or near heavy traffic areas.

Analytical investigations on the program show that the capability to collect relatively large masses of samples, free from troublesome substrate interference, greatly facilitates obtaining complete compound identification of inorganic and organic species. It is recommended now that basic analytical methodology developments be carried out to reduce compound identification methods to routine practice in order that large numbers of samples can be processed economically. This would permit the obtaining of far more comprehensive and meaningful data bases than are available at this point for promulgating air quality standards, prescribing control process limitations, and assessing health burdens of particulates.

Prior to extensive field collection and analysis efforts, it is recommended that the precision, collection efficiency, and study of possible sulfur dioxide (SO_2) and nitrogen dioxide (NO_2) reactions during sample collection be studied in greater depth. The possibility of ozone (O_3) formation in the electrostatic section should be checked more thoroughly, although no O_3 odor can be detected and the overdesign feature permitting operation at about 8000 volts appears to preclude this concern. The collection efficiency for very small aerosols, estimated 50 percent for the 0.05-μm range, should be checked.

SECTION III

INTRODUCTION

Each year the evidence demonstrating health effects of pollutants has become more impressive, and there now is little debate on whether pollutants are harmful. Rather the amount and types of pollutants which are safe and tolerable have become the subject of debate.

During recent years it has become apparent that the total mass concentration of particulates is not a definitive measure of the health hazard due to airborne particles. A major reason for this is that usually the aerosol sampling has been performed using single stage collectors, with these collections analyzed to determine the mass concentrations and/or the constituents of the overall samples. Since most aerosols are polydisperse, the mass median size approaches that of the larger particles in the sample. Thus, the mass concentration values of such samples are for practical purposes determined by the concentration of the relatively fewer larger particles. If these particles are nonrespirable, the measured mass data will have little direct relationship to the inhalation hazard of the pollutants, and attempts made to correlate health effects with particulate burden can be ineffective.

Size selective particulate sampling and analysis obviously would be more meaningful, but thus far most sampling of this type has been directed toward obtaining information primarily on particle size distribution. The sample mass obtained for each size range is quite small and is entirely inadequate for complete chemical and physical characterization. A further problem often posed both by single and multistage samplers has been that of the substrate collector stage or stages. Frequently the collected sample cannot be separated

totally from the collector substrate, and the resulting substrate contamination severely impairs the accuracy of analysis of the collected particulates.

The program, defined in concert with the EPA Project Officer and described in the following sections of this report, was designed to develop a system which would overcome the present day obstacles in obtaining size selective particulates on a substrate interference-free basis in sufficient quantities to provide the comprehensive analyses needed to realistically identify pollutant characteristics with health effects studies.

OBJECTIVES

The overall objective of this program is to determine the chemical composition of airborne particulate matter which constitutes the respirable size in humans. In order to achieve this objective, a sampler must be designed capable of:

- Collecting large masses of particles in the respirable range and excluding particles above the respirable range, and
- Utilizing a collection system or medium which will not alter the particulate matter collected and which will yield a sample in a form and size (mass) amenable to a wide array of analytical methodology development effort.

The primary objective of the study is to obtain elemental and compound composition of those species which may have a deleterious effect on human health with particular attention to be focused on the sulfate species (both organic and inorganic).

PROGRAM PLANNING

SAMPLER DESIGN

The deposition site of particulate matter in the lung is a function of particle size, particle density, and breathing rate. Larger particulates which manage to penetrate beyond the nasal-pharnyx region deposit on the

ciliated epithelium of the upper trachael-bronchial tree and effectively eliminated from the lung within a few hours. Smaller particles deposit in the lower portions of the lung which are not ciliated and may remain in intimate contact with the thin alveolar membrane for weeks or months (depending upon solubility). Therefore, it is essential to know what portion of an air pollutant falls in the size fraction which will deposit on the nonciliated portion of the lung.

When particle size data are missing on total particulate loading obtained on an absolute filter, the NCRP-ICRP standards* have provided a built-in particle size correction which is based upon the assumption that only 12.5 percent of the weight of the aerosol reaches the nonciliated portion of the lung. Therefore, it is necessary to develop samplers which will eliminate the need for this arbitrary assumption that 12.5 percent is respirable. Actually when the primary source of the aerosol is from a combustion source, a major portion of the aerosol is respirable. Small differences in mass median diameter of an aerosol can produce profound differences in the region of prominent deposition.

It was the concept of this part of the study to develop a hi-vol sampler that has sampling characteristics which duplicates the "Respirable Dust Standard" defined by the Health and Safety Laboratory (HASL).

The desired sample size of at least 1 g requires that the sample flow rate be extremely large for most ambient conditions in order to collect this sample within a reasonable period of time (1 or 2 days). The power limitation -- 15 amperes at 110 volts with a high efficiency blower which will pull about 5 inches of water vacuum -- limits its size to about 30 cubic meters per minute.

The restriction that not more than 1 percent of the sample collected can be larger than 8 μm while maintaining an effective cutoff size of approximately 3.5 μm appears to require the use of an impactor stage rather than a high-volume cyclone. (Subsequent to the initiation of the experimental program, the requirements of the sampler design were changed to provide for a two-stage

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*ICRP Standards: "Particulate Deposition and Retention Models for Internal Dosimetry of the Human Respiratory Tract," Health, Vol. 12, pp. 173-207, 1966. (Task Group on Lung Dynamics, International Commission on Radiological Protection.)

collection which would accommodate collection of secondary aerosols. (Details of this provision and design changes are given in the Experimental Work section of this report.)

SAMPLE ANALYSIS

Plans for complete chemical composition analyses usually divide samples into two major classes: organic and inorganic. Within these major classes there is a further subdivision: elemental and species or compound identification which must be considered if appropriate data for health effects are to be obtained. In planning the analysis of collected samples it is anticipated that relatively large masses of each particulate size fraction will be available so that, for the most part, separate sample portions can be used for each analytical procedure or method. The general scheme planned for chemical and physical characterization of samples is shown in Figure 1. The organic soluble fraction methods of analysis, gas chromatography (GC), mass spectrometry (MS), GC-MS, and infrared (IR) give compound and/or species class indentification. This is not true for the inorganic compounds. Generally, complete elemental analyses were planned using such techniques as optical emission spectrography (OES); X-ray fluorescence (XRF); spark source mass spectrometry (SSMS); atomic absorption spectrometry (AAS); gas mass spectrometry (GMS); anion determination; ultimate analyses (CHN); total sulfur and carbon; plus possible oxygen

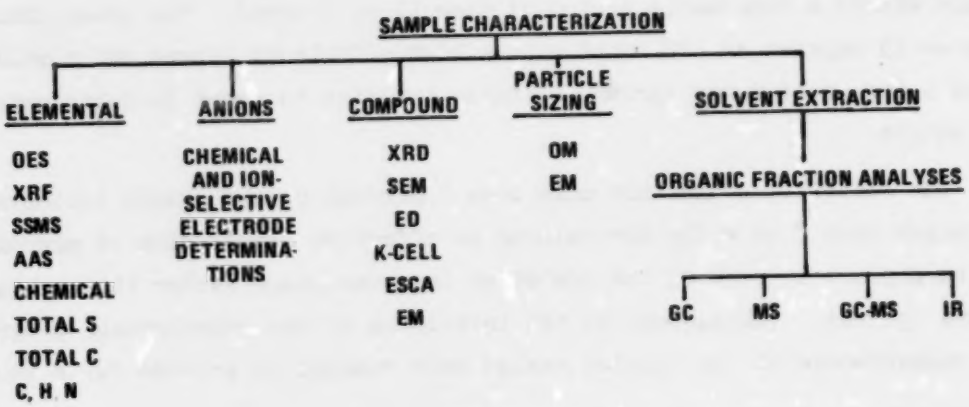


Figure 1. Generalized analytical scheme.

determinations. These should give a reasonable material balance on the samples. Determination of inorganic species or compounds are much more difficult. Available for these data are X-ray diffraction (XRD); scanning electron microscopy (SEM); electron diffraction (ED); electron microscopy (EM); Knudsen cell mass spectrometry (K-cell MS); and electron spectroscopy for chemical analysis (ESCA).

Originally, ten sets of duplicate samples from selected urban areas were planned in this program. On revision of the planned sampler design, this was reduced to two to three areas to accommodate the increased effort required to redesign and construct a three-stage sampler system rather than the originally proposed two-stage. Finally, only single site sample collections were undertaken in the program, since it appeared to be of most need to utilize the samplers immediately to collect particulate at the EPA Background Catalyst Study site.

EXPERIMENTAL

Shortly after the inception of the program, discussions with the EPA Project Officer revealed that there were some questions among the health study group at EPA and elsewhere on the exact particle size fractions of particulates which would represent the most realistic sample collections for health studies. Pending resolution of this problem, initial efforts on the sampler design and construction were carried out on a small prototype system.

INITIAL SAMPLER DESIGN CONSIDERATIONS FOR A TWO-STAGE SAMPLER

Initial design considered a two-stage, hi-vol sampler which would provide a collection efficiency of the first stage to duplicate the Health and Safety Laboratory (HASL) or "Los Alamos" curve as shown in Figure 2. Both an impactor stage and a bank of miniature cyclones were considered to remove the large diameter aerosol particles. An impactor stage should have a cutoff size between 3.5 and 4 μm. The impactor jets would be extremely long slits and the velocity through the slits would be minimized to reduce particle blowoff on the impaction surface and to reduce the overall pressure drop.

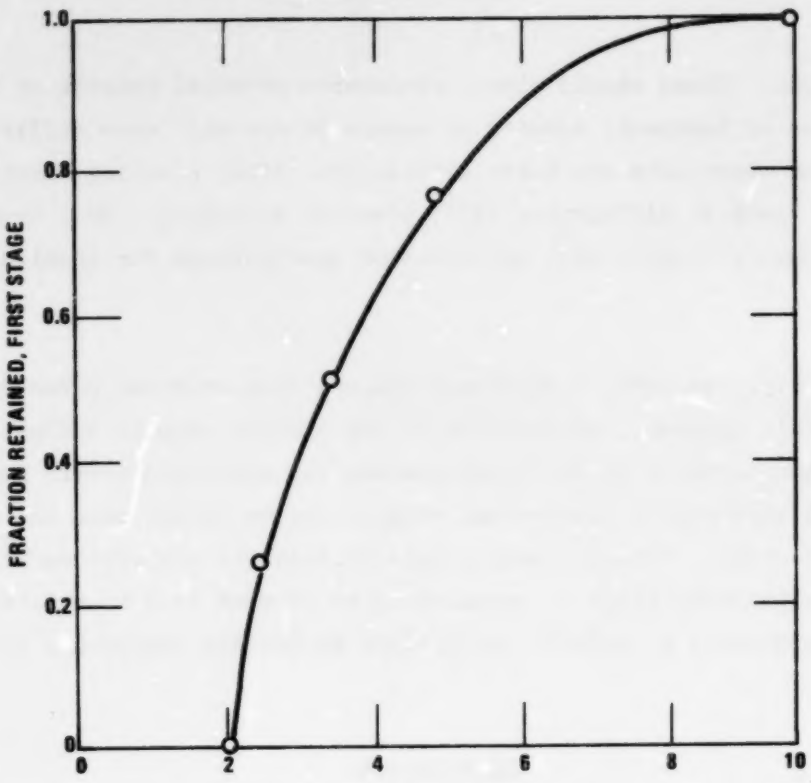


Figure 2. Los Alamos Curve for first stage collection.

If it were found that the cyclones had the desired operating characteristics, they would be used since they can be purchased commercially. Dr. Morton Lippman* has shown that the Aerotec 2 cyclone, when operated at the proper flow rate, closely reproduces the HASL curve. Figure 3 obtained from Mr. Lippman's final report (December 15, 1972) shows the composite collection curve efficiency obtained for four Aerotec 2 cyclones at several flow rates. The curve obtained at a flow rate of 450 liters/min comes closest to reproducing the HASL curve. It would be expected the the larger Aerotec 3 cyclone

*Lippman, M., "Evaluation of Three Cyclones for AEC-AGIN Size Selective Sampling." Final Report on Contract HSM099-17-48, NIOSH.

would have a similar collection efficiency at about the same pressure drop. Although the cyclones did appear to provide the desired characteristics, consideration of their use subsequently was abandoned since a large number of units would be required to obtain the flow rate needed for large mass collection which in turn would make the sampler unwieldy from a bulk and weight aspect.

A high-efficiency electrostatic precipitator appeared to be most suitable for collection of the very fine respirable-size particulates on the basis of collection efficiency, low pressure drop, and minimal substrate problems. Filters would have to be massive in size in order to avoid an excessively high pressure drop and would create an undesirable substrate problem. An impactor stage, even with extremely long slits, would not be efficient for particles

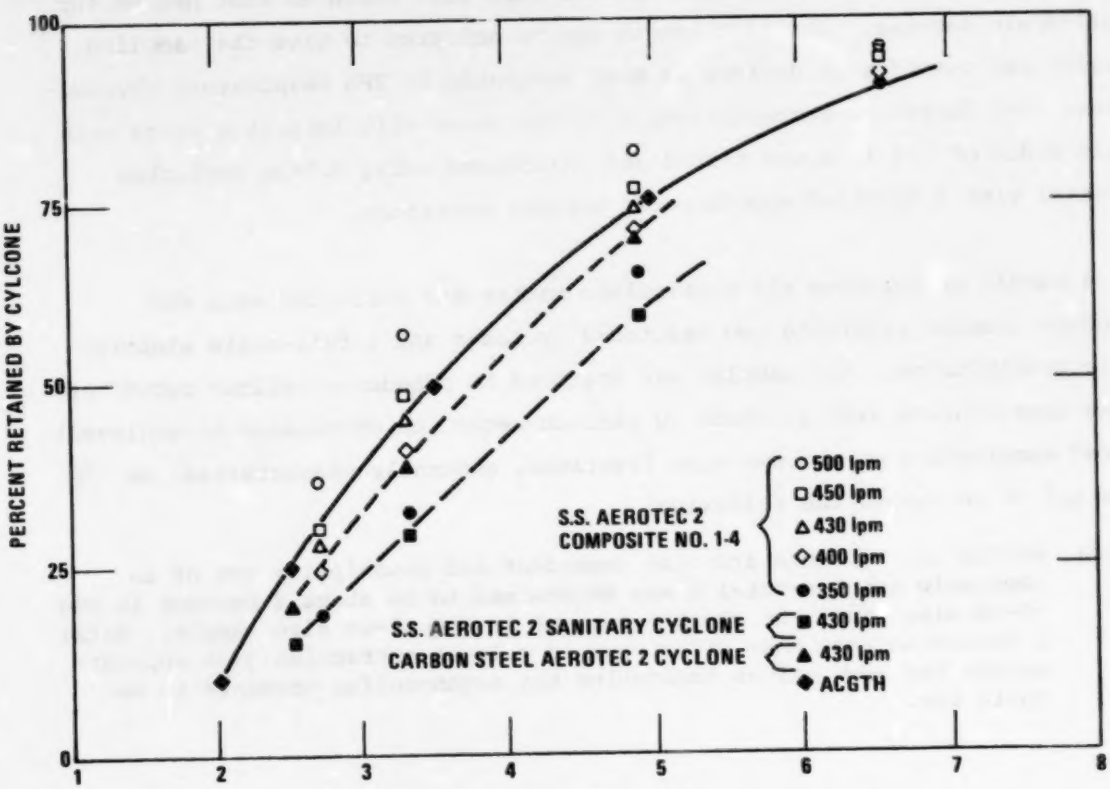


Figure 3. Collection efficiency of Aerotec 2 cyclones.

less than 0.5 μm. The electrostatic precipitator necessarily would require special design to provide for high efficiency for particles less than about 0.5 μm and to minimize the possibility of O_3 formation which could cause chemical reaction during the collection process.

TWO-STAGE PROTOTYPE SAMPLER

A small-scale prototype sampler using two Aerotec-2 cyclones and a full-scale electrostatic sytem was assembled to test the collection efficiency for respirable range particulates. This small unit has the capacity (not fixed) of providing the size distribution desired by EPA and can be scaled up to provide the requisite sample mass.

In addition to this assembly, a small-scale (approximately 5 cfm) impactor stage was constructed. This stage has the same slit width as that needed for a full-scale sampler. Its slit length can be adjusted to give the sampling capacity and cutoff size decided as most desirable by EPA respiratory physiologists. The impaction characteristics of the large slit impaction stage with a slit width of 1/4 inch was tested and calibrated using 3.5-μm particles generated with a Berglund monodisperse aerosol generator.

A sample of Columbus air particulate matter was collected with the prototype sampler utilizing two Aerotec-2 cyclones and a full-scale electrostatic precipitator. The sampler was operated to produce a cyclone cutoff of 3.5 μm (particulate size at which 50 percent detection efficiency is achieved). Limited examination of the two size fractions, generally characterized as >5 μm and <5 μm showed the following:

(1) Sulfur (S) contents are size dependent and principally are of an inorganic nature--total S was determined to be about 2 percent in the >5-μm size range and about 6 percent in the <5-μm size sample. Total S determinations made after organic solvent extraction gave approximately the same values indicating the organosulfur contents to be quite low.

(2) The electrostatic precipitator fraction was extremely difficult to recover. This suggested a redesign wherein the plates can be individually removed for cleaning and where a precipitator substrate material which will not materially contaminate the collected sample is used.

Resolution of Definition of Respirable Range Particulates

In meetings with EPA aerosol physiologists and leading experts* in aerosol sampling technology, it was decided that:

(1) The ACGIH curve provides the best knowledge to date as to what constitutes the respirable aerosol fraction and that the first stage of our sampler should try to duplicate this curve as much as possible and have a cutoff size of 3.5 μm.

(2) Secondary aerosols (principally photochemical) are less than 2 μm, and a special stage should be added to our sampler to collect a major portion of these aerosols. It was decided that the cutoff size for this stage should be close to 1.5 μm.

Revised Sampler Design - Three Stage Collector

The cutoff size of the second stage of the sampler was made at 1.7 μm. The slight reduction to 1.5 μm would have reduced sampling rate considerably compared to only about a 25 percent reduction for a 1.7-μm stage.

In the design of the sampler, special consideration was given to the best means of collecting the first two size fractions. For the first stage either cyclones or impaction techniques could have been used with reasonable power requirements. The problem with cyclones is that the collection efficiency curves begin to deteriorate at the higher flow rates. At least 30 cyclones would have been required to maintain our sampling rate and still have a reasonable collection efficiency curve. The size of the sampler would have been increased quite dramatically using cyclones.

The addition of the intermediate stage for the collection of the primary respirable aerosols would almost necessitate using impaction techniques.

*A list of attendees at this meeting is given in Appendix B.

Cyclones to achieve the desired particle size classification would have extremely low flow rates and excessive power requirements. The size of the sampler would have been increased significantly and the sampling rate decreased drastically.

Based on the above, it was decided to use impaction stages to obtain the desired particle collection. Impaction characteristics are much better predicted than sampling characteristics of cyclones. Also, impaction plates are much easier to fabricate.

The requirement that there should be minimum contamination with the collection substrate almost dictated that the submicron particles be collected using an electrostatic precipitator. It would be virtually impossible to remove these particles from any filter media, and, at a rate of 1.3 million cubic feet per day, the quantity of filter media required would be unfeasible. In the design of the electrostatic precipitator it was decided to make two compromises.

(1) The voltage was to be maintained at less than 8000 volts in order to minimize O_3 formation.

(2) The precipitator was to be overdesigned in order to compensate for the low voltage, yet maintain a high collection efficiency.

The precipitator was designed so that the plates could be removed for cleaning. Also they were constructed of high purity Al in order to minimize sample contamination. The impactor plates were Teflon coated to reduce any possibility of sample contamination.

Figure 4 is a schematic drawing of this sampler. The figure shows the scalping stage to remove the extremely large particles, the two impactor stages and the high efficiency electrostatic collector.

The sampler utilizes inertial impaction to obtain the desired size classification. Both the primary cutoff size of 3.5 μm and the secondary size of about 1.7 μm use multiple long slits for the plate-type orifice. The slits

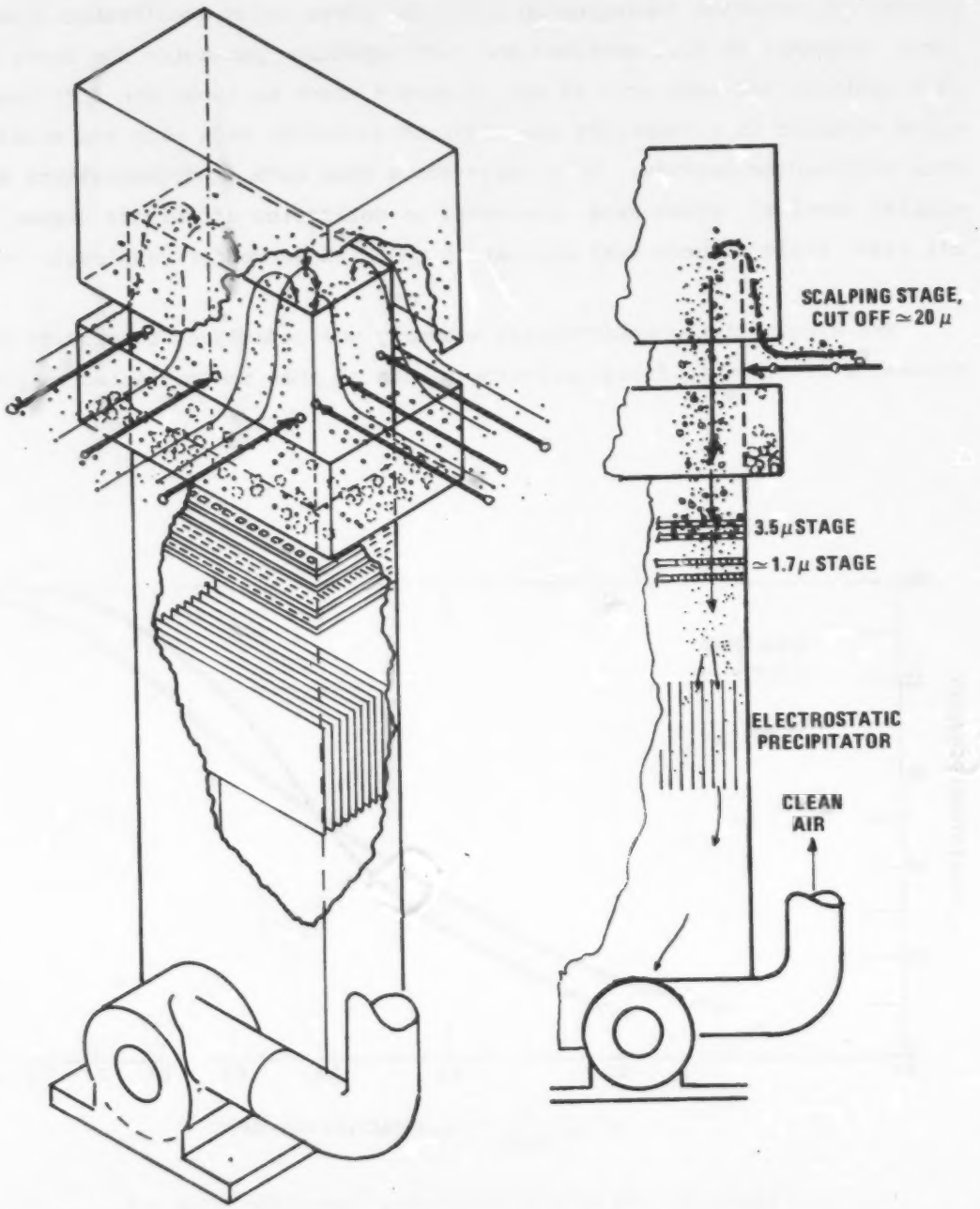


Figure 4. Prototype of high-volume air sampler.

are 1/4 x 1 inch and 1/16 x 1 inch, respectively. Calibration tests show that the impaction parameter $\Psi^{\frac{1}{2}}$ for 50 percent collection efficiency is equal to 0.39 for both slits using the desired cutoff size of droplets of unit density. (The symbol $\Psi^{\frac{1}{2}}$ is a dimensionless parameter which characterizes the impaction process of particles impinging on a surface after being accelerated from a jet.) Gussman, et al., obtained the same impaction parameter for Stage 2 of a slit width of 1/4 inch or 0.63 cm. Figure 5 shows an impaction efficiency curve obtained in calibrating the 1/4-inch (0.63-cm) wide slit and similar data obtained by Gussman. This curve shows that both experimentalists achieved similar results. These data, presented as collection efficiency versus particle size, yield a curve very similar to the HASL curve for respirable aerosols.

The electrostatic precipitator assembly was redesigned to provide for removable rather than fixed collector plates so that the collected sample can

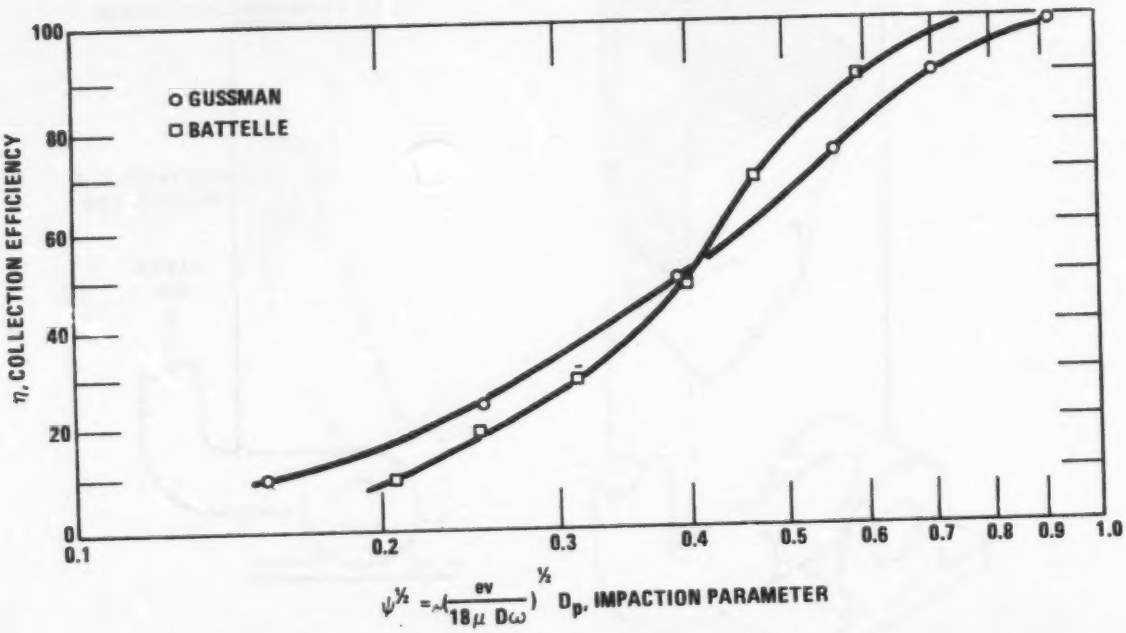


Figure 5. Impaction efficiency curve for slot jet.

be removed more readily. Very pure Al (99.99 percent) was selected as the optimum material for the precipitator collection plates. The selection of Al was based largely on the factors that Al can be obtained in pure form and the possible small contamination incurred in removing particulate collections from the Al substrate should not materially affect the analysis. The possible choices of noble and precious metal surfaces were rejected due to costs and because of the EPA interest in monitoring catalytic converter exhaust emission pollutant species.

As designed, the full-scale samplers occupy a floor space of about 32 x 48 inches with a power requirement of 110 volts and 20 amperes.

Construction of Samplers, Calibration and Laboratory Trials

With preliminary calibrations completed, detailed layouts and engineering drawings were made for the samplers' construction. Two were built. Detailed engineering drawings and a list of purchased items are to be included in a companion article to this publication. This subsequent publication presents revisions to the sampler and final commentary, suggestions and instructions for its construction. The samplers were assembled and tested. The design characteristics were for a total pressure drop of about 3 1/2 inches of water through the impactor stages. This, coupled with pressure drop through the entrance and exit, should have produced a total pressure drop of 5 inches at a flow of about 900 cfm using a 1-hp high efficiency blower. On test it was found that the total Δp was 6 3/8 inches with over half produced by the exit plenum. Flow straighteners were built in to eliminate this defect, and the total Δp was 4 7/8 inches at cfm.

The samplers were challenged using a fluorescent aerosol with a mass mean diameter of 6.5 μm. It was found that 90 percent of this aerosol deposited on the first stage and the penetration through the electrostatic precipitator stage was less than 0.01 percent.

Initial laboratory tests of the samplers showed good mechanical and electrical component reliability. In later testing high voltage leaks occurred

in the electrostatic precipitator section. After checking, this was attributed to moisture pickup of the red fiberboard insulating material used to hold the electrostatic plates in place. This material was replaced with Teflon, and on further testing no further leakage problems occurred.

Field Site Application

The initial field site application of the sampler(s) was carried out during the period September 6 through 13, 1974, at the Los Angeles Catalyst Study (LACS) site. Two samplers were taken as cargo on the same commercial air flight as carried the Battelle sampling and engineering team. Those were set up the same day as departure at Sites A (upwind) and C (downwind). They were operated at approximately road level.

After a preliminary 2-day run to observe loadings obtained at these sites, a 4-day sampling effort was performed. During that period the particulates were removed from the first and second impactor stages each day. The particulates on the electrostatic plates were allowed to accumulate for the entire 4-day period and subsequently were removed at Battelle Columbus Laboratory (BCL). The approximate masses obtained during this period are given in Table 1.

SITE LOCATION	SIZE FRACTION	TIME PERIOD	MASS, g	PERCENT
SITE A (UPWIND)	3.5-20 μm (1ST STAGE)	~4 DAYS (100 hr)	1.440	25.5
	1.7-3.5 μm (2ND STAGE)	~4 DAYS (100 hr)	0.845	14.9
	<1.7 μm (ELECTROSTATIC PLATES)	~4 DAYS (100 hr)	3.370	59.6
TOTAL			5.655	100.0
SITE C (DOWNWIND)	3.5-20 μm (1ST STAGE)	~4 DAYS (100 hr)	1.245	18.6
	1.7-3.5 μm (2ND STAGE)	~4 DAYS (100 hr)	0.945	14.2
	<1.7 μm (ELECTROSTATIC PLATES)	~4 DAYS (100 hr)	4.495	67.2
TOTAL			6.685	100.0

Table 1. Particulate collection at L.A. site.

The aerosol at both sampling sites of the massive volume sampler was sampled with a special cascade impactor developed at BCL for sampling automotive exhaust. This impactor has a sampling rate of 1.0 cfm and extremely sharp impactor stages of 0.25, 0.50, 1.0, 2.0, 4.0 and 8.0 μm. The collections were made on 2 consecutive days (not concurrently) and covered approximately a 24-hour period for each run. The particle size data for this impactor as well as that obtained from the 100-hour sample collected in the massive volume impactor are shown in Figures 6 and 7. These data show that the particle size data obtained by the two impactors agree and that the downstream aerosol is slightly smaller than the upstream aerosol. It also shows that between 70 and 80 percent of the particulates are in the respirable size range.

Vapor phase (chromosorb bed) samplings also were made at each site for about a 24-hour period each--again, not concurrently. Analyses of these would be significant and enlightening for organic compounds which to a certain extent could be "washed off" the particulates during samplings. However, investigation of these vapor phase collections were beyond the scope of this program.

The two samplers were operated continuously from the period September 6 through early November, 1974. During this period, EPA technicians at the Catalyst Site were instructed to remove the collected particulates each day from each of the two impactor stages of each sampler. This procedure should require no more than a 15-minute period of shutdown for each sampler. The electrostatic precipitator should have the capacity to contain particulates from up to a month or longer collection time depending on the aerosol concentration. The removal of particulates from the electrostatic precipitator plates requires 4 to 8 hours, although it appears that the process could be automated by use of an ultrasonic bath.

During the continuous 24-hour operation over a 2-month period, several problems, both of an operational and of a sample recovery nature, were observed. These were resolved under a program modification effort described later in this report.

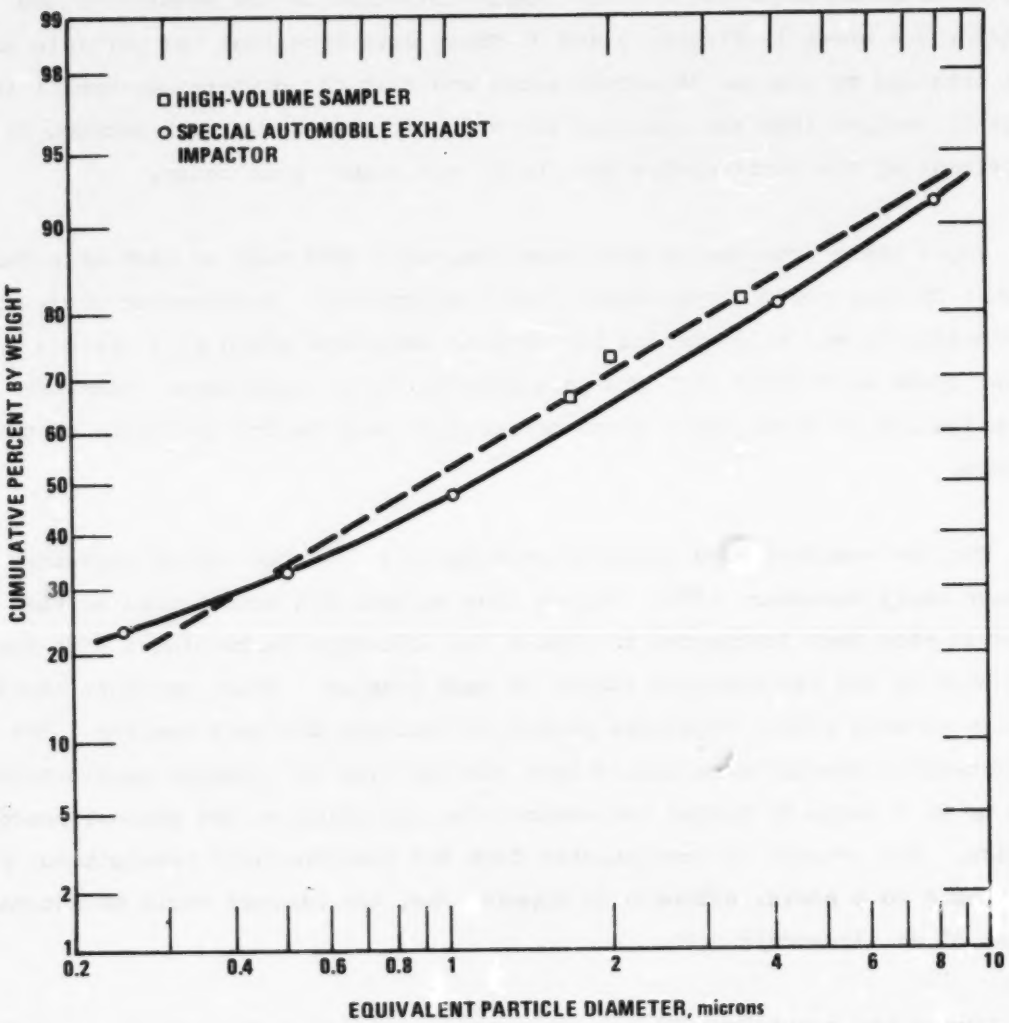


Figure 6. Particle size distribution of downwind pollutants at San Diego freeway site.

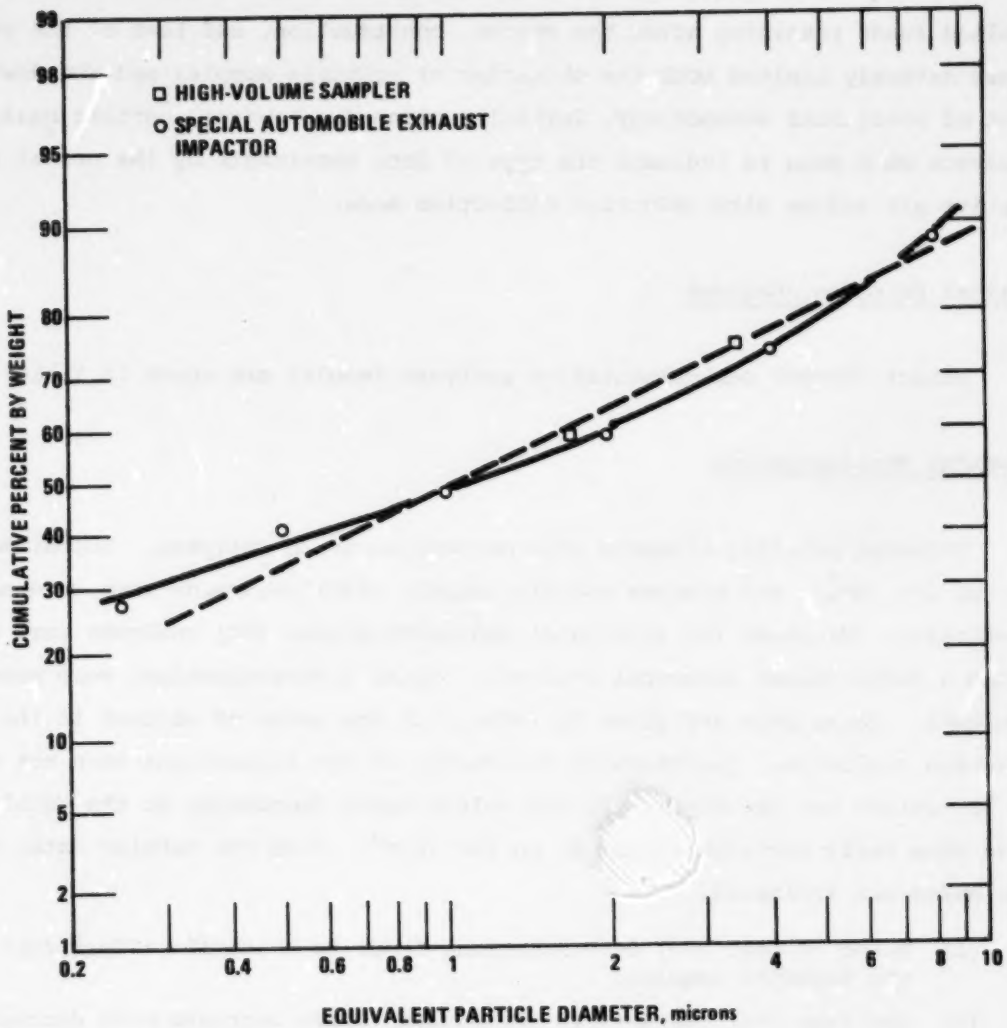


Figure 7. Particle size distribution of upwind pollutants at San Diego freeway site.

SAMPLE ANALYSIS

The request to place the two samplers at the LACS Background Site and the limited funds remaining after the design, construction, and test of the samplers severely limited both the obtaining of multiple samples and the development of analytical methodology, initially planned. However, certain basic analyses were made to indicate the type of data obtainable by the use of a massive air volume size-selective collection mode.

Optical Emission Analyses

Direct current semiquantitative analyses results are shown in Table 2.

Chemical Determinations

Selected metallic elements were determined by AA analyses. Anion, Ammonium ion (NH_4^+), and benzene soluble organic (BSO) determinations were made chemically. Nitrogen (N) (ultimate) and hydrocarbons (HC) analyses were made using a Perkin-Elmer Elemental Analyzer. Total N determinations were made by Kjeldahl. These data are given in Table 3 on the basis of percent in the fraction collected. Quantitative recoveries of the collections were not made so TSP values are not given. Hi-vol values taken frequently at the Catalyst Site show daily variations from 60 to 150 $\mu g/m^3$. From the tabular data, the following are indicated:

(1) Based on Lead (Pb) determinations there is a traffic effect seen in the downwind samples.

(2) The Iron (Fe) contents in the several sizes decrease with decreasing size. Similar decreases were found by optical emission spectrography for several other elements, generally of crustal origin.

(3) S and BSO, on the other hand, increase in concentrations in the finer size fractions.

(4) Sulfate ($SO_4^=$) accounts for most of the total S in the two larger size fractions. The $SO_4^=$ to S ratio plus the total S versus S after benzene extraction indicate the possibility of organo sulfur compounds accounting for up to 1 to 2 percent of the total S in the <1.7- μm fractions.

SAMPLE DESIGNATION	ELEMENTS DETERMINED, percent																
	Al	Ba	Ca	Cr	Co	Cu	Fe	Pb	Mg	Mn	Ni	K	Si	Na	Ti	V	Zn
UPWIND																	
1ST STAGE	10	0.01	5	0.01	<0.01	0.5	5	1	3	0.1	<0.01	3	15	2	0.5	0.02	0.05
2ND STAGE	5	0.01	5	0.01	<0.01	0.5	3	1	3	0.1	<0.01	3	15	2	0.5	0.02	0.05
ELECTROSTATIC	5	<0.01	1	0.02	<0.01	1.0	1	1	1	0.1	<0.01	3	10	2	0.5	0.03	0.1
DOWNWIND																	
1ST STAGE	10	0.01	5	0.01	<0.01	0.5	5	1	3	0.1	<0.01	3	15	2	0.5	0.02	0.05
2ND STAGE	5	0.01	5	0.01	<0.01	0.5	3	3	3	0.1	<0.01	3	10	2	0.5	0.02	0.1
ELECTROSTATIC	5	<0.01	1	0.02	0.01	1.0	2	3	1	0.2	0.01	4	10	2	0.5	0.05	0.1

Table 2. Semiquantitative optical emission analyses for metals.

SAMPLE DESIGNATION		DETERMINATION										
		Pb	TOTAL S	Fe	BENZENE-SOLUBLE ORGANICS	$S^{(a)}$	$SO_4^{=}$	C	H	N	NO_3^{-}	NH_4^{+}
SITE A UPWIND	1ST STAGE	0.86	2.0	3.6	8.0	2.4	6.45	7.3	1.5	2.2	7.75	0.87
	2ND STAGE	1.13	5.6	2.9	9.0	6.5	16.8	7.9	2.4	2.5	2.85	2.55
	ELECTRO-STATIC	0.93	8.7 [8.9] (b)	1.0	16.6	9.4	21.3	6.5	4.3	3.1	6.5	3.55
SITE C DOWNWIND	1ST STAGE	2.43	2.0	3.8	4.3	1.9	6.3	8.6	1.7	1.9	7.4	0.69
	2ND STAGE	3.16	4.7	3.1	8.8	5.2	14.6	10.6	2.3	2.3	5.5	2.17
	ELECTRO-STATIC	3.74	5.5	1.9	16.0	6.1	17.4	9.4	3.4	2.5	3.0	2.45

(a) AFTER BENZENE EXTRACT.

(b) RECHECK DETERMINATION.

Table 3. Analyses of collected particulates from L.A. background catalyst study site - results in percent.

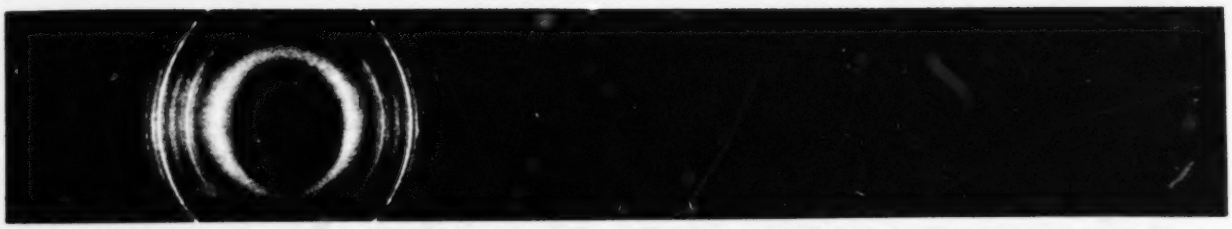
(5) The nitrite (NO_3^-) form accounts for ~70 percent of the total nitrogen (N) found in the large particle size while NH_4^+ accounts for ~75 percent of the total N found in the fine fractions.

X-Ray Diffraction

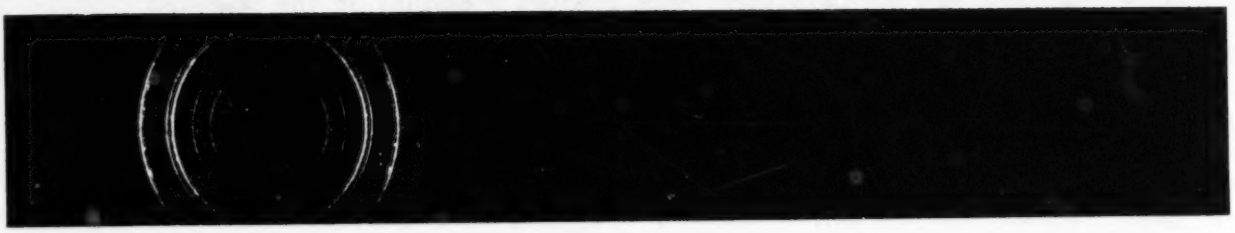
Diffraction patterns were obtained by the first stage (>3.5 μm) and electrostatic precipitator (<1.7 μm) fractions both upwind and downwind. The patterns are shown in Figures 8a, b, c, and d. Detailed data interpretations of the patterns were not made. However, pattern differences can be detected visually. Note the greater complexity of the fine versus the coarse particle size fractions and the relatively more complex structure of the downwind fractions. Because of the complexity of air particulate compositions, full exploitation of the XRD technique generally would require the use of fractional separation techniques prior to taking of the patterns and computer data search. Organic fraction removal, magnetic fraction separation, water-soluble fractionation and density separations are among the techniques which can be used to obtain more definitive diffraction data. The availability of large sample amounts permits this type of exploration to be done while still having adequate sample for other compound identification efforts.

Organic Species Identification

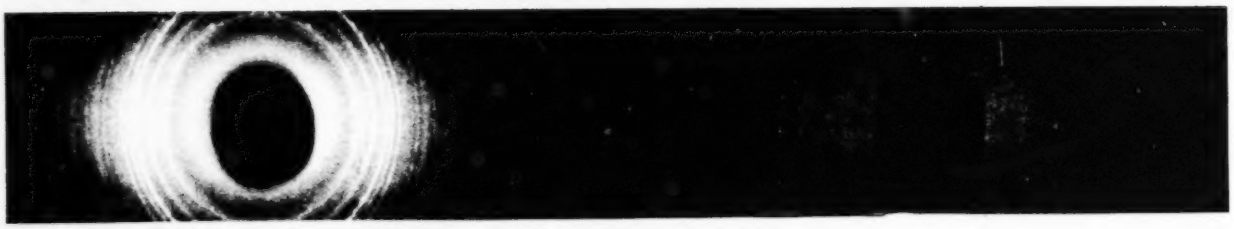
Each of the three fractions of Santa Monica Freeway particulate was subjected to methylene chloride extraction, and a preliminary survey for an appropriate GC column was carried out. It was quickly evident that exceedingly complex mixtures of organic compounds were present, and that it may not be feasible to attempt a comprehensive analysis on one GC column alone. A polar high resolution column (25 ft, 3 percent Silar 5CP) was chosen as being the most suitable for resolution of most aliphatic and smaller aromatic materials. It is anticipated that PNA species can be analyzed without difficulty using a Dexil 300 column preceded by isolation of the species of interest by the Rosen liquid chromatographic separation.



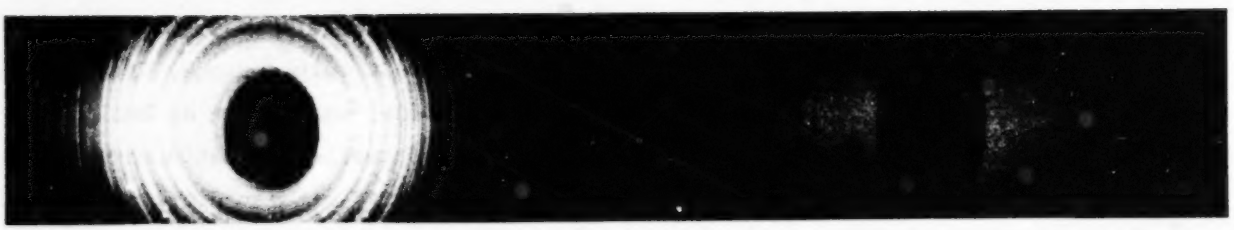
A. UPWIND >3.5 μm FRACTION



B. UPWIND <1.7 μm FRACTION



C. DOWNWIND >3.5 μm FRACTION



D. DOWNWIND <3.5 μm FRACTION

Figure 8. X-ray diffraction patterns of 4-day (100 hr) collections.

Preliminary analyses indicate that the great majority of organic materials are found in the two smallest size fractions. These latter fractions exhibit many similarities in organic composition, although there do appear to be several possible significant differences. The largest size fraction (>3.5 μm) is quite different from the smaller two, and shows none of the low molecular weight polar species which they exhibit. An envelope of unresolved high molecular weight compounds is common to all these fractions, but is more abundant in the small size fractions. This unresolved envelope appears to consist of a highly complex mixture of approximately C_{12} to C_{30} aliphatic HC isomers.

Gas chromatographic mass spectrometric analysis was carried out using both electron ionization and chemical (methane) ionization spectra--the chemical ionization spectra almost always give invaluable molecular weight data, and the characteristic fragmentation is often sufficient to make a reliable molecular assignment when coupled with GC data. Electronization could be additionally carried out in order to gain further structural data from the more extensive fragmentation.

Considerable spectra detail was obtained, but an intensive spectral matching computer search was not completed. The spectra have been reserved for interpretation under an anticipated extension of the present program.

SAMPLER AND COLLECTION PROCESS MODIFICATIONS

The two Massive Air Volume Samplers proved suitable in their performances during tests carried out in the laboratory and during the initial 2-day and 4-day field tests at the LACS Site. During these periods the samplers were operated and the particulate collection was recovered by Battelle personnel. As stated earlier, the program plans initially had called for relatively short-term (days) sampling at various CHESS sites by Battelle personnel and detailed analyses of the collected samples.

The unplanned requests that the samplers be left at the Background Catalyst Study Site for continuous (years) 24-hour operation by technicians

not familiar with the sampler construction, assembly detail, and sample recovery process brought about needs to improve the structural stability of the samplers and to modulize the collection assemblies. These needs were discussed with the EPA Project Officer and increased time and funds were approved for the following changes.

(1) Detailed engineering changes were made to provide for additional structural stability to the Impactor Plate Assembly and Electrostatic Plate Assembly holders. The changes accommodated for both materials of construction changes and machining and assembly tolerances.

(2) Four Impactor Plate Assembly holders were constructed using Al metal coated with Teflon. These replaced the two holders presently in place in the samplers (one in each) made from red fiberboard construction. The availability of two holders for each sampler would provide a simplified and contaminant-free mode of replacing the two impactor plates and two impactor slit stages when they are loaded daily with collected particulates. The sampler operator simply can remove the impactor plate holder module which contains collected particulate and slide in a fresh unit to provide for essentially continuous operation.

(3) Two additional electrostatic plate assembly holders were constructed using strengthened design features. The two present holders in the samplers were similarly strenghthened in design. Again, the availability of modulized replacement of particulate-laden plates with cleaned plates would ensure contamination-free handling and provide essentially continuous collection.

(4) Two freight shipping boxes for the electrostatic plate holder assembly and plates were constructed. Since the electrostatic plates (47 in each sampler) can accumulate up to 1 month of urban aerosol sampling and since the particulate removal from the same 100 ft^2 of collector surface is lengthy and tedious, it was deemed useful to be able to ship these to a clean, well-equipped laboratory for sample recovery, cleaning, and subsequent reuse in the sampler. With the shipping boxes the plates could be air freighted to Research Triangle Park, North Carolina, Battelle, or elsewhere.

(5) Air volume flow and electrostatic precipitator meters were installed for ready check on the samplers performance.

SECTION IV

DISCUSSION

Based on the results achieved at this stage, the program efforts show that a long-needed sampling and analysis technology can be fulfilled. Through collection of a large mass of sample on a size basis, thorough characterization of respirable range particulates can be made and thus good correlations between particulate burden and health effects are attainable. The applications of the system are many. The samplers could be utilized at the several CHESS sites where the health effects of populations are already under study. Point source emissions could be monitored by taking upwind and downwind samples. If the sampler developed under this program, together with a thorough analytical survey of the collected respirable size fraction, were coupled with a gas phase sampling and analysis system it would be possible to obtain a total knowledge of the airborne health threat posed to a given population group. Such knowledge is needed for useful and meaningful health/pollution burden studies.

The design and construction aspects of the program entailed greater efforts than originally anticipated. This coupled with an urgent need by EPA for the samplers to be placed at the Background Catalyst Study Site precluded the obtaining of more thorough data on collection efficiency and precision as well as checking the possibility of chemical reaction due to O_3 formation in the electrostatic precipitator section of the sampler.

Additionally further isolation and identification of suspect compounds are needed. The identification and exploration of pathways to quantitative assessment of the oxidized sulfur species which give rise to adverse health

effects have been correlated with sulfate ion concentrations found in the water extract of particulate matter collected on glass-fiber filters. These identifications have been and remain a primary target for our investigations.

APPENDIX A

OPERATIONAL AND PROCEDURAL DESCRIPTION OF MASSIVE AIR VOLUME SAMPLER

GENERAL DESCRIPTION OF MASSIVE AIR VOLUME SAMPLER

The Massive Air Volume Sampler is designed to pull a very large volume of air, ∿100 ft^3/min, through the sampler system and to collect particulates in three size ranges. The first two size ranges of 3.5 to 20 μm and 1.7 to 3.5 μm are collected by a two-stage impactor system housed in an impactor assembly module, and the third size range, <1.7 μm, is collected by a high-efficiency electrostatic precipitator. In use, for urban aerosol collection, the particulates should be removed from the impactor plates daily and from the electrostatic plates about monthly. These time periods will vary depending on the particulate burden of the air being sampled.

Viewing Figure A-1, the air inlet is through the 2-inch slot (covered by a fine mesh screen to repel insects) seen near the top of the assembly. The impactor assembly is in a drawer, closed by snaplocks on either side of the sampler just below the air inlet. (Only one upper snaplock can be seen in the Figure.) The electrostatic assembly is located just below the impactor assembly. Again, it is closed by snaplocks, only one of which can be viewed in Figure A-1. The motor and motor housing is in front of the sample collector with the egress air outlet seen pointing left. The intake to the motor housing, of course, must be closely fitted to the sampler collector so that no air loss occurs between the sampler and the motor housing. A flow meter and electrostatic voltage meter are provided as a check on the operational performance of the sampler (Figure A-2).

Figure A-1. Overall photograph of massive air sampler.

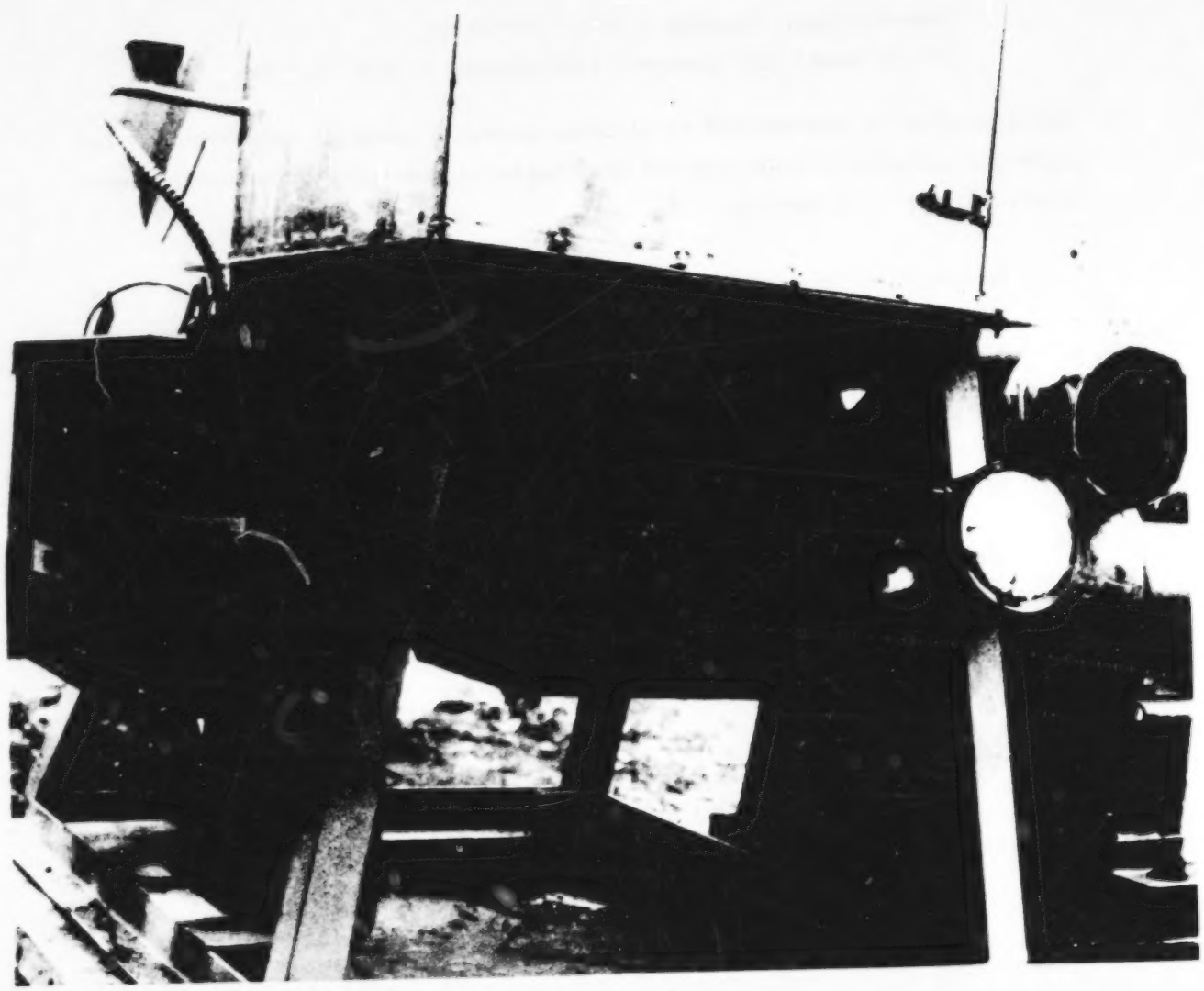


Figure A-2. Lower part of massive air sampler showing flow meter, electrostatic power unit, and voltage meter.

The particulate collections of course should be kept separate when recovered. They should be placed in large opaque containers (Teflon is preferred) according to stages:

First Stage, Impactor - Size 3.5-10 μm

Second Stage, Impactor - Size 1.7-3.5 μm

Third Stage, Electrostatic Precipitator - Size <1.7 μm.

Refrigeration is recommended to minimize possible chemical interaction. Two impactor assembly modules and two electrostatic precipitator assemblies are provided with each sampler.

In operation the sequence of sample collection is as follows:

(1) Shut off power to the motor blower and to the electrostatic precipitator--generally every 24-hour period.

(2) Remove the impactor plate assembly from the sampler and take to a clean area or place in the impactor assembly shipping box.

(3) Install a second impactor plate assembly having cleaned (particulate removed--not washed) plates.

(4) Start up the sampler again.

(5) In a clean area, remove the collected particulates from the impactor plates and replace the plates in the assembly for reinstallation and collection in the sampler. The objective of this operation (which is repeated daily) is to prevent excessive particulate buildup on the impaction plates which could be blown off resulting in improper size fractionation. Do not wash or excessively scrub the particulates from the plates. Repeated collection and removal will result in nearly total recovery over a time period.

(6) The electrostatic assembly sample recovery should follow the same general procedure as above, except the assembly will be changed only about once a month. The recovery of particulates from the 47 plates or about 100 ft^2 of surface will require up to an 8-hour period. It is recommended that at least each month the electrostatic assembly containing the loaded plates be placed in the shipping box provided and be sent to a well-equipped clean laboratory for particulate recovery, reinstallation of the 47 plates, and reshipment to the field collection site.

OPERATIONAL GUIDE

Setup of Sampler System

It is assumed in this description that no electrical connections have been made and that the impactor plate assembly and electrostatic precipitator assembly are not in place in the sampler.

(1) Select a location to provide intake air at the desired vertical level. Keep in mind that the egress air flow is of very high volume and may resuspend particulates which could get into the ingress air stream. A large sheet of plastic spread below the sampler base can minimize this problem. In Figure A-1, the sampler and blower are seen fastened to a large sheet of plywood.

(2) The base on which the sampler and blower motor are placed should be approximately level. The weight of the sampler and blower motor is about 500 pounds.

(3) Secure the sampler and blower motor independently to the base to minimize movement during operation. There are bolt holes provided for this as seen in Figure A-2. Prior to fastening, be certain that the blower motor housing inlet is in alignment and snugly fitted to the bottom sampler air fitting. The sampler should also be positioned so that the high velocity blower exhaust is not deflected back into the sampler inlet.

(4) Install impactor plate assembly (Figures A-3 and A-4). Open the two snapfasteners located near the top of the sampler and raise the door opening. Remove the impactor assembly from shipping box and carefully slide the impactor assembly (Figure A-3) into the opening. NOTE: The impactor assembly may be already in place in the sampler. In that event it may be useful to check that the four plates are correctly oriented as follows:

(a) The impactor assembly consists of a unit Teflon "box" containing four sets of machined grooves into which are fitted four Teflon-coated (green) stainless steel plates. It is important that these plates be in the right sequence and proper alignment, both in the initial setup and in subsequent collections. The proper impaction plate sequence is as follows:

Plate	Position[(a)]	Slit Width, in.	Purpose
1	Top	1/4	Stage 1 Jets
2	2nd	3/8	Stage 1 Impaction Plates
3	3rd	1/16	Stage 2 Jets
4	Bottom	1/8	Stage 2 Impaction Plates

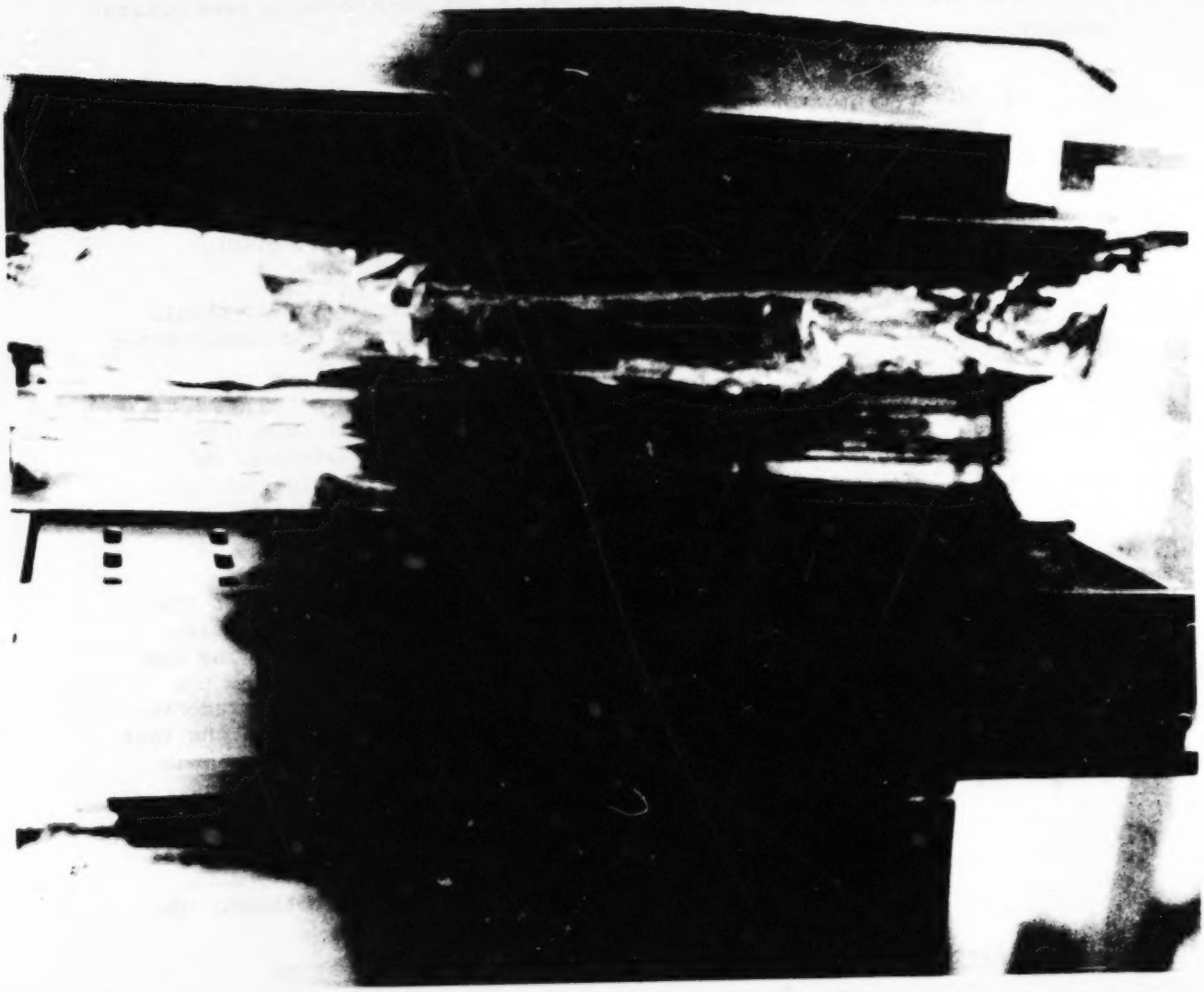


Figure A-3. Impactor plate assembly module, partially withdrawn.

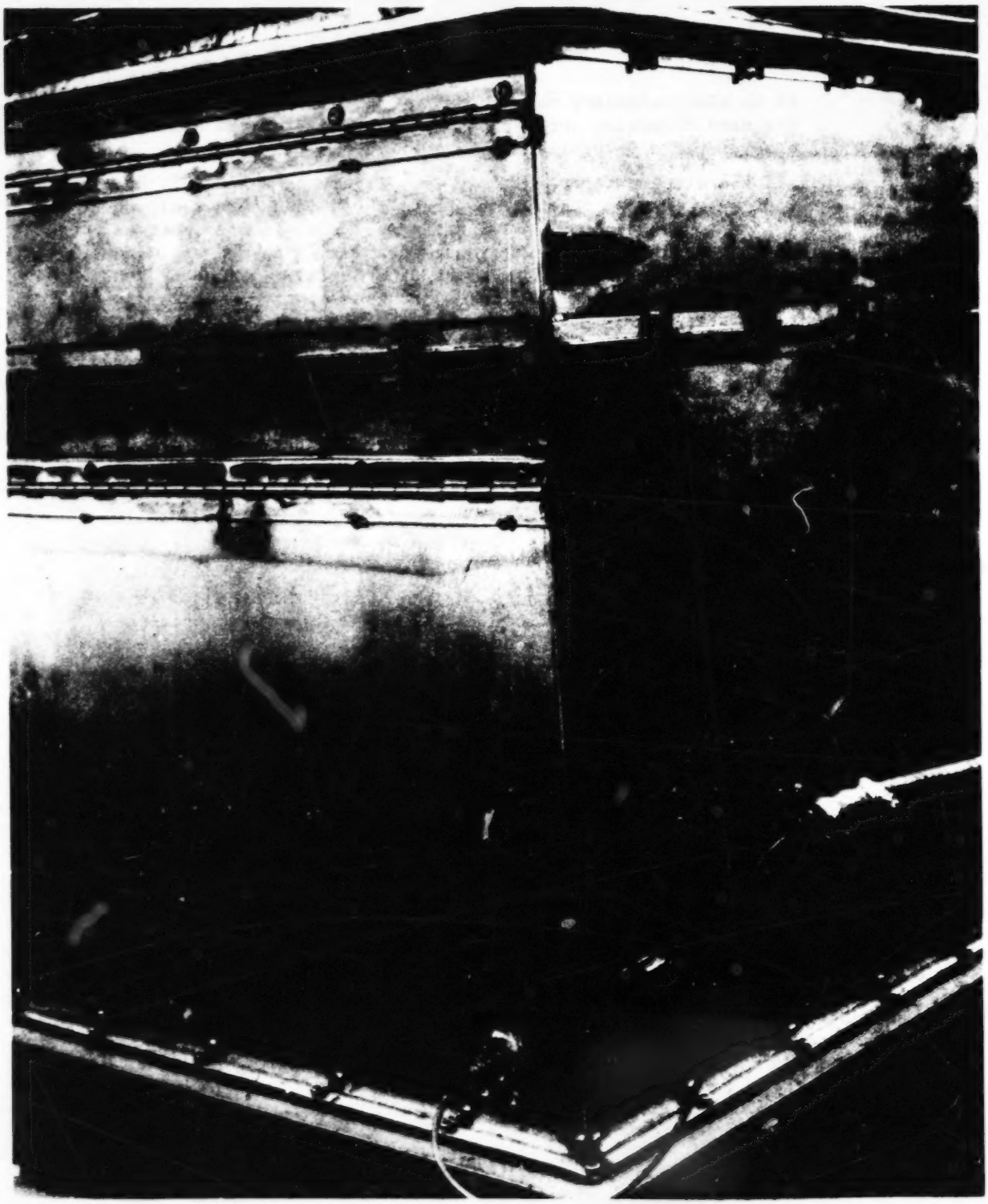


Figure A-4. Closeup of impactor plate assembly (upper) and electrostatic precipitator (lower) closed drawer compartments.

It is also necessary that the slits for all four plates run in the same direction (preferably perpendicular to the axis of the electrostatic precipitator plates).

(5) Install the electrostatic assembly (see Figures A-3 and A-5) by (a) similarly opening the two snaplocks located on either side near the bottom of the sampler, (b) opening the precipitator door, and (c) carefully sliding the electrostatic assembly into place. Again, the assembly may already be in place. Do not lift using the handle seen on the front. It will not support the weight of the precipitator plate assembly. The purpose of the handle is to be able to pull the precipitator plate assembly out of the sampler far enough so that it can be lifted from underneath. After the precipitator plate assembly is placed completely within the sampler, it will then be possible to close the precipitator door and lock it with the two snap connectors.

(6) The electrostatic precipitator plate assembly has a male banana plug which extends through the precipitator door. High voltage is supplied to the precipitator plates by means of an insulated female portion of the plug which is connected to the power supply. Connect the male and female plug together.

(7) After completion of the above steps the sampler is ready to be connected to an electrical outlet. It is recommended that both the blower and precipitator be connected to a 100 volt duplex plug from a single outlet with a capacity of at least 15 amps. It is also recommended that an electric clock be plugged into the same circuit in order to check on electrical outage.

(8) After both the blower and precipitator power supply have been connected, check to ensure that both are functioning properly. The switch on the precipitator power supply should be turned on and the voltmeter should read approximately 800. (Actual voltage to the precipitator plates is about 7800 as the voltmeter has a voltage divider with a factor of 10 located in its input.) If the voltmeter reads substantially below 800, there is a short in the system--either the precipitator plate assembly or the ionizer section. These can be isolated by disconnecting the banana plug at the precipitator door. The problem should be readily discernable as the high voltage will arc at the trouble point. The high volume blower will essentially provide a constant volume flow through the sampler unless there is an extremely low voltage input. A magnehelic pressure gauge is attached to the blower to indicate total flow and should read at least 5 inches of water.

(9) Daily Change of Impactor Assembly and Recovery of Impactor Collections.* Remove the entire Impactor Assembly unit by opening the snaplocks at the top of the sampler and opening the door. Using the knurled screws, partially withdraw the unit until it can be grasped at the bottom with two hands for final withdrawal. Take to a clean area for sample recovery. (NOTE: The second impactor assembly can be put in place and the Sampler system restarted.)

Figure A-5. Electrostatic precipitator assembly, partially withdrawn.

Using a large Allen wrench remove the four Allen-head screws seen at the four corners of the assembly--Figure A-4. Disassemble and clean the plates in the following sequence:

(a) Top Plate. Using plastic throw-away gloves, withdraw the top slot guide plate and place on a clean sheet of aluminum foil. There should be little if any collection on this guide plate. Brush any particulate onto a small square or tray of aluminum foil and then place the collection, if any, into a sample collection bottle labeled "1st Stage." Set the cleaned plate aside for subsequent reinstallation (in the same position and slot orientation) in the Assembly unit.

(b) Second Plate (1st Stage Collector). Repeat as described above. This plate should contain a fairly even load of particulates. Brush and/or loosen lightly using a Teflon coated knife or tool. There is no need to excessively scrape or abrade to obtain a total 100 percent recovery since the operation will be repeated approximately daily. Recover the particulates and place them in the sample bottle marked "1st Stage." Place the plate aside for reinstallation.

(c) Third Plate. This is the slot plate for the second impactor stage assembly. It should contain very little collection. Treat as with (a) above but place any collections in a sample bottle marked "2nd Stage."

(d) Fourth Plate. This plate contains the second stage impactor collections. Treat as with (b) above but add the collections to the sample bottle marked "2nd Stage." Reinstall the four plates in the order and orientation they were removed. (See Instruction 4 above.)

(10) Bimonthly or Monthly Change of Electrostatic Assembly.

(a) Turn off power supply switch and disconnect electrical connection (banana plug) to electrostatic precipitator. (See Figure A-3.) Open the two snaplocks and raise the metal drawer door. Using the pull handle on the front of the assembly (Figure A-5) partially pull the assembly out to the point where the assembly can be lifted. Do not lift using the pull handle.

(b) Place the Electrostatic assembly with the particulate-laden plates in the shipping boxes provided.

(c) Install the second Electrostatic assembly; connect the electrostatic electrical power connection (banana plug) and turn on power supply switch.

(11) Recovery of Sample Collection from Electrostatic Plates. Open the shipping box and remove the electrostatic assembly in a clean area. Remove the plates individually, place on a clean subsurface such as aluminum wrap and remove the collected particulates by scraping both sides of each plate with a sharp-edged Teflon spatula. Using a clean lab brush, brush the particulates into a sample bottle labeled electrostatic precipitator catch.

After recovery of the sample, scrub the plates clean using a detergent and fine abrasive powder, rinse with deionized water, dry, and reinstall the plates back into the electrostatic assembly according to the following procedure.

The precipitator plate assembly is designed so that every other plate is either at ground potential or connected to the high voltage potential. Thus, in order that the individual plates can be interchangeable they are notched at the bottom corner, so that there will not be a direct short across the two buss bars. The first plate should be installed so that the unnotched corner slides into a small clip attached to the buss bar and the notched corner does not touch the buss bar on the opposite side. Each additional plate is placed in the same manner and the notches will be opposite each other.

After all the plates have been installed, place the assembly into the shipping box and ship back to the sampling site.

APPENDIX B

LIST OF ATTENDEES OF AEROSOL SAMPLING TECHNOLOGY MEETING

A meeting was held December 11, 1973, at Research Triangle Park to discuss, review, and obtain a consensus of opinion from EPA personnel and leading experts in the field on the proposed sampler designed to duplicate the HASL or ACGIH collection efficiency for respirable particulates recommended by the ACGIH Threshhold Limits Committee. The curve has been well defined by respiratory physiologists and has a cutoff size (50 percent collection of 3.5 μm). The design was considered within the parameters of collection of gram quantities of ambient air particulates in a 24-hour period with minimal substrate interference, low power requirements, and portability. The meeting was attended by the following:

Name	Affiliation
Morton Lippman	New York University
Harry Ettinger	Los Alamos Science Laboratory
T. T. Mercer	University of Rochester
R. J. Thompson	EPA
Elbert Tabor	EPA
J. H. Knelson	EPA
A. V. Colucci	EPA
S. Simione	EPA
Bob Burton	EPA
Walter Crider	EPA
W. Wilson	EPA
W. Cary Eaton	EPA
R. I. Mitchell	Battelle Columbus Laboratories
W. M. Henry	Battelle Columbus Laboratories